Effect of Water Soluble Polymers on Physicochemical Properties of Soil and Soil-Amino Acid Interactions

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by

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Certificate

I hereby declare that the short synopsis **"Effect of Water Soluble Polymers on Physicochemical Properties of Soil and Soil-Amino Acid Interactions"** being submitted in partial fulfillment of the requirement for the degree of Doctor of Philosophy in Chemistry under FET, of Manav Rachna International Institute of Research & Studies, Faridabad during the academic year 2022-2023. It is a bonafide record of original work carried out under the supervision of Dr. Shagufta Jabin, Professor, Dept. of Applied Sciences, FET, MRIIRS, that has not been submitted in full or part to any other university or institute for degree or diploma. Further, it is certified that short synopsis is plagiarism free and proof has been submitted.

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ABSTRACT

The enormous increase in the world population so far has resulted in an increase in global food demand. In comparison to 2021, agricultural productivity is expected to improve by at least 50% by 2050. To achieve this, new agricultural practices will be required. Water soluble polymers have recently been used in agriculture to demonstrate several benefits, including soil amendments, soil stabilization, water conservation, reduced soil nutrient consumption, curtailing the negative effects of moisture stress in crops and dehydration, minimizing the eutrophication and water contamination caused by fertilizers and pesticides and controlling several phytopathogens. Water soluble polymers are widely used in many industries, including paper, water, food processing, mineral processing, textile, medicines, surface coating, petroleum production, oil and natural gas recovery, etc. Water soluble polymers from different kinds of industries are generally disposed of in sewage or wastewater treatment systems. The ultimate goal of soil management is the protection of the environment in a manner commensurate with public health and socio economic concerns. The aim of the research is to study how different water soluble polymers, adding to a soil, can change the properties of the soil as well as the interaction of the soil with amino acids.

Keywords: Organic polymers, physicochemical properties, soil amendments, soil-quality improvement, water soluble polymers.

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1. Introduction

"Soil" comes from a Latin word, "solum" which means ground (Khanifar, 2021). It plays a vital role in supporting life in the universe. “Soil Chemistry” is an interesting branch from a perspective of research, as on one hand it has a unique characteristic of producing and sustaining crops, but on the other hand, there are some soils which may be unable to produce any kind of crop, nevertheless they are soils (Hartemink, 2006). The productivity of soil depends upon the physical and chemical properties of soil itself (Budak and Calisir, 2019). By the term physicochemical properties of a soil, we mean by some soil parameters like mechanical composition, pH, electrical conductivity (EC), temperature, moisture-content of both wet and dry soil, exchangeable cations and cation exchange capacity (CEC) (Kekane *et al.*, 2015). The chemistry of soil can be understood by studying different soil characteristics which include color of soil, soil structure, soil-texture, particle-size distribution, bulk density, soil fertility, adsorption property, water stability of soil, etc. (Embe and Asriani, 2021).

Some of the physicochemical properties have been explained below to develop a better understanding of properties and structure of soil.

Physicochemical Properties of Soil

Mechanical Composition

It is the very basic and preliminary information required about a soil. It indicates the presence of different sized particles such as gravel, sand, silt and clay in soils (Dazhou, 2021). The chemical physical and biological properties of soil are influenced by the particle size distribution (PSD). Out of gravel, sand, silt and clay, clay particles have a large surface area and are responsible for determining most of physicochemical properties of the soil. The presence of particles of different sizes is also responsible for influencing the water holding capacity, which is one of the various characteristics of soil. The strength and compressibility of soils are also affected by the particle-size distribution (Shipton and Coop, 2012).

Soil pH

The soil pH indicates, in a rough manner, “an active fraction of the hydrogen ions present in the soil phase”. The acidity and basicity of the soil is indicated by the soil pH. At pH 7, $[H^+] = [OH^-]$, where $[H^+]$ = hydrogen ions’ concentration present in the soil and $[OH^-]$ = hydroxide ions’ concentration present in the soil. The acidic range of soil is from pH=0 to pH=7 and the alkaline range is from pH=7 to pH=14. Soil pH= -log $[H^+]$. The minerals’ solubility in the soil and the plant

growth are greatly affected by the soil pH (Gentili *et al.*, 2018). The activities of useful microorganisms are also influenced by the concentration of hydrogen ions or hydroxide ions in the soil (Neina, 2019). For instance, legume bacteria, responsible for nitrogen fixation, are less active in soil with high [H^+], while on the other hand fungi usually tolerate the acidic environment of soil in a better manner. Plants which tolerate the acidity of soil and grow well in the environment are pineapple, tea, blueberries etc. in contrast, beans, barley and sugarcane grow in a well manner in somewhat acidic to adequate basic soils (Kidd and Proctor, 2001).

Soil Temperature

The temperature of soil varies from -20 degree Celsius to 60 degree Celsius. The physical, biological and chemical properties of soil are influenced by the soil temperature. The microorganisms are biologically active in warm soil rather than in cold soil (Pietikäinen *et al.*, 2005). The soil surface has a different temperature than that of the inner layer of soil.

Electrical Conductivity (EC)

This physicochemical property of soil tells about the presence of total soluble salt in the soil. Higher the salinity of soil, more will be the soil EC (Bauder *et al.*, 2014). When E.C. is greater than 4.0 mmhos per cm, the soil is considered to be saline enough to indicate the existence of NaCl and Na_2SO_4 as soluble salts in soil (Grieve and Shannon, 1998).

Exchangeable Cations

The exchange of A^+, a positive ion by B^+, another positive ion is called exchangeable cation. Ca^{2+}, Mg^{2+}, H^+, Na^+, K^+ and Al^{3+} are the most common cations which are present on the exchangeable sites in soil (Pansu and Gautheyrou, 2006). But the addition of gypsum to the soil can constantly change the amounts of these cations on the colloidal surfaces (Favaretto *et al.*, 2008). The plant nutrients such as magnesium, calcium and potassium are supplied to the plants in the form of exchangeable cations. The cation exchange sites on the soil surface adsorb positively charged metal ions such as Cd^{2+}, Zn^{2+}, Ni^{2+}, Pb^{2+} etc. from the industrial wastewaters (Garcia Sanchez, 1999). The amount of cations in the soil solution are closely related to the exchangeable ions, which can be changed by the variation in the concentration of a cation in the colloidal solution.

Cation Exchange Capacity (CEC)

CEC is well-defined as the quantity of exchangeable **c**ations present in each unit weight of dry soil. It also refers to the interaction of the soil with cations in a way that they can be exchanged by the other cations on simple leaching instead of getting removed from the solution completely. The clay component of soil is known to interact or combine with the cations so well that the CEC is high for

the soil with high amounts of clay particles (Lagouin *et al.*, 2021). The change in a pH can result in the change in the CEC also.

Characteristics of Soil

Soil Structure

The organization and arrangement of particles of soil comes under the category of soil structure (Nayaka *et al.*, 2021). All the processes of soil including chemical, physical and biological processes are controlled by soil structure. Soil particles have variations in size and shape. Due to this variation, a soil structure could not be determined in an exact manner. According to soil scientists, soil structure is a storehouse of carbon and atmospheric carbon dioxide (Ontl, 2012). According to the literature the soils' physical properties are correlated with PSD (Particle Size Distribution). Soil has different particles with the given sizes viz.; macro-aggregates (> 250μm), micro-aggregates (20μm -250μm), silt (2μm – 20μm) and clay particles (< 2μm) (Shi *et al.*, 2021).

Soil Color

Soil color and presence of elements in the soil are inter-related to each other. For example, soil with cobalt content is blue/red in color, presence of lead makes the soil look yellow in color, and white color of soil indicates the presence of calcium in it and so on (Han *et al.*, 2016). One can also predict the age of soil by noticing its color (He *et al.*, 2003). The color of soil has also a strong relationship with the physicochemical properties.

Soil Texture

Soil Texture is determined by the three basic components of soil i.e. sand, silt and clay. Their sizes come under the particle-size distribution, another soil characteristic. Among the three components of soil, clay has an ability to shrink and swell because of evaporation (Pooni *et al.*, 2019), irrigation and plant water uptake. This property of clay further results in the change in the texture of soil.

Particle-size Distribution

It is a vital property of soil which is used for the analysis of mole fraction of particles of silt, sand and clay present in the soil. Various direct and indirect methods of measurement are there, like it can be measured directly with the help of a microscope and diffraction method is an indirect method used in its measurement (Abbireddy and Clayton, 2009). Sieve analysis is a technique used for the separation of soil particles and analysis of their sizes (Díaz-Zorita *et al.*, 2002).

2. Literature Review

Review of literature gives an essential understanding of the methodologies that were used in the past.

2.1. Soil-Amendments

The physicochemical properties of soil and soil characteristics get affected by various soil amendments which result in contamination of soil either directly or indirectly. Different additives from different industries can be taken into the account which include soil amended with pesticides, soil amended with fly-ash, soil amended with organic acids, soil amended with alcohols, soil amended with water-soluble and water-insoluble polymers, soil amended with surfactants, soil amended with dyes and many more (Antonious, 2016).

The role of such amendments is not only to modify the physicochemical properties and composition of soil but to affect the mobility or interaction of some organic and inorganic matter with the soil as well.

2.2. Water soluble polymers (WSPs), as Soil-Conditioners

Water-soluble polymer (WSP) is also called water-soluble resin (WSR). It can be dissolved in water and is able to form the solution or dispersion liquid. WSP is also known as a hydrophilic polymer material due to the presence of a macromolecular long-chain along with the hydrophilic groups like carboxyl, hydroxyl, amide, amine, ether etc. (Rivas *et al.*, 2009). They tend to possess various important properties like film formation, adhesion, dispersion, flocculation, etc. due to the hydrophilic nature of them (Guo *et al.*, 1998). In the field of soil and water conservation, WSPs are gaining attention to a greater extent. They are acting as soil conditioners due to the various remarkable properties and hydrophilic nature. Soil conditioner is a chemically modified natural substance used to stabilize the texture of the soil, favorably modulating the physical or structural properties of the soil (Reddy *et al.*, 2013).

WSPs are likely to be disposed of either into a public or private sewage/wastewater treatment system. After use, WSPs have a tendency to get eliminated from various industries into the aquatic environment such as streams, rivers, lakes etc. In such environment, a WSP may remain soluble or may be adsorbed onto the solids that remain suspended in the treatment systems (Swift and Baciu, 2006). If it passes directly into the aqueous environments or gets adsorbed on the suspended solids both raise the same issues to the environmental health. Adsorption of a WSP on sewage sludge in treatment systems leads to other disposal possibilities such as land filling, composting,

incineration, and as a fertilizer for soil as soil amendments (Pilavtepe *et al.*, 2018). The fate of WSPs have been discussed, in a pictorial form (Figure 1), after getting eliminated from various industries and factories.

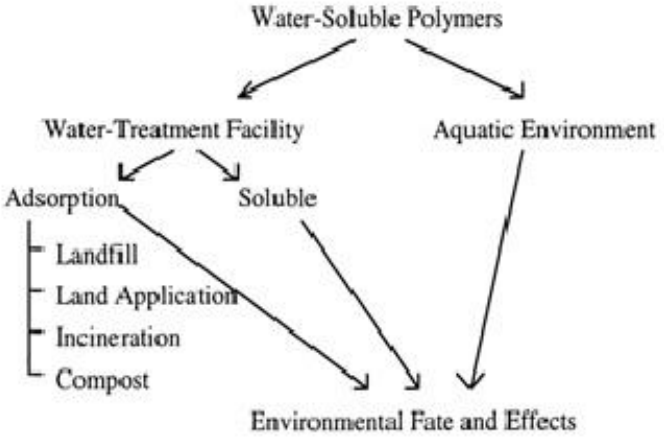

Figure 1. Major Disposal Options for Water-Soluble Polymers (Swift, 2002)

WSPs are classified into three on the basis of the functional group carried by them. These are cationic, anionic, or nonionic in nature, this classification affects their uses and a characteristic to biodegrade also. On the basis of occurrence, WSPs are further classified into three categories, natural, synthetic and synthetically amended natural polymers (Kadajji and Betageri, 2011). The synthetic WSPs may be further divided into addition and condensation polymers, where in addition polymers, there is a presence of carbon-chain backbone mostly while in condensation polymers a heteroatom chain is present as a backbone of the polymer. PAA and PVA viz. Poly(acrylic acid) and poly(vinyl alcohol) respectively are the examples of addition polymers, known as functional polyolefin also. On the other hand, polymers including polyesters, polyamides, and polyether are considered to be condensation polymers, which are related to many other naturally occurring polymers in terms of similar structures and hence are fantastic source for the synthesis of synthetic water-soluble biodegradable polymers. Overall, WSPs can be categorized in a different ways, in terms of molecular weight, functional charges (cationic, anionic and nonionic), hydrophilic-hydrophobic compositional balance, structure (branched, linear, cross-linked, etc.), and synthetic or natural origin. Properties of both the polymer and soil are known to influence the degree of interaction between the two. As it is mentioned in the particle distribution of soil, it has three components: sand, silt and clay and polymers interact with the clay particles of the soil mostly.

Properties of polymer including polymer configuration, nature, molecular weight, amount of surface charge and size are some of the important properties of polymer which affect the interaction of the polymer with the soil (Móczó and Pukánszky, 2008). In the similar manner, there are few important properties of soil also such as soil solution ionic strength, type and amount of clay, with which the polymer interacts mostly, soil pH and nature of ions in soil solution. Type of charge on polymer affects the adsorption of polymer on clay fractions of soil in a great manner. The order followed by polymers in terms of adsorption on soil is cationic > nonionic > anionic. Nonionic polymers show interaction with soils through Vander Waals interactions. High molecular weight polymers get adsorbed on clay surfaces by uncoiling and spreading themselves over the surfaces. In this process, there is a replacement of the water molecules adsorbed in a double layer around the clay particles of the soil. Cationic polymers are absorbed on anionic soil constituents through electrostatic force of attraction. As a result a reversal of surface charge takes place. Adsorption of anionic polymers take place through "polymer bridges" between negatively charged groups of soil and negatively charged group. The role of the cation bridge is to bridge together the polymers and the negatively charged soil constituent together. “The degree of anionic polymer adsorption was dependent on the type and amount of exchangeable cations, amount of clay, pH, and polymer molecular size” (Ben-Hur, 2006).

Wind and water erosion cause 75 billion metric tons of soil loss each year in the world, so erosion control technology has received great attention. The application of Polyacrylamide (PAM) is a reliable technology for soil and water conservation (Sojka *et al.*, 2007). Due to its water solubility and versatility, PAM has been successfully used as a soil conditioner in earlier times. It is a synthetic organic polymer with a high molecular weight and water solubility. Polymer lengths can be extended by 6 million monomer units. It is used for strengthening paper and board, as a binder and clogging agent for oil recovery, as a soil stabilizer, thickeners and in textile applications. Paper industry is the second largest user of PAM after the wastewater treatment plant. PAMs can be neutral, cationic and anionic polymers. The amount of its surface charge that can be generated varies by hydrolysis, which together with the pH of the solution affects the 3-dimensional configuration of the polymer and therefore of the adsorbed polymer. The degree of hydrolysis of PAM can be changed by replacing $-NH_2$ with -OH in the polymer, this is called hydrolysis, forming an anionic polymer. The degree of hydrolysis affecting the adsorption of PAM to soil may be due to the change in polymer configuration as reported.

Polymer bridging plays a key role in PAM/soil adsorption considering that anionic PAM combines

with soil, which is generally negatively charged colloidal particles (Green and Stott, 1999). In polymer bridging, a divalent cation such as metal interacts with anionic moieties from both PAM chains and soil. Polymer bridging is understood by the fact that the efficiency of anionic PAM in reducing erosion losses increases with the addition of calcium or any other metal, which acts as a cationic bridging in anionic PAM interaction with negatively charged particles of soil. The anionic PAM with 20% hydrolysate was reported to be the most effective in soil stabilization providing the most extended chain configuration for polymers for adsorption. There is a greater absorption of PAM in the illiterate than in montmorillonite (Ben-Hur, 2006). During PAM interaction with montmorillonite, the adsorption efficiency decreased as the anionic properties of PAM increased. On the other hand, the reverse case is observed for PAM-illite solutions. This may be because the adsorption of PAM occurred on the outer surface of the clay, and the structure of the clay fragments (micro particles), formed in the presence of PAM and influenced the adsorption of PAM polymers. The structure of these micro particles is different for different clays and hence the adsorption of PAM. Adsorption on both clays is increased in the presence of electrolyte concentrations in solution, which supports the fact that cations are involved in polymer adsorption to the soil.

Since the 1990s, PAM has been widely studied for reducing soil erosion. In addition, the viscosity of water increases after PAM is applied, which leads to reduced water flow and better water retention (Sojka *et al.*, 2007). Anionic PAM is known to prevent the formation of a seal on the soil surface and reduce soil loss. PAM stabilizes the soil structure as well as decreases the hydraulic conductivity of the surface by preventing the formation of surface seals (Sojka *et al.*, 2007). Due to Coulomb force and van der Waals force, PAM particles flocculate in water with sufficient electrolyte to attract soil particles and increase aggregation. It also shows that PAM is highly effective in soils with high clay content because its molecules are adsorbed to clay minerals by forming bridges with exchangeable multivalent cations (Mamedov *et al.*, 2007). PAM penetrates as aggregates and makes the outer surface stable, as a result stabilizing the soil aggregates (Wu *et al.*, 2012).

PAM is a powerful technique for erosion control. During irrigated agriculture, to reduce erosion and improve infiltration. It has been demonstrated that it can successfully stop furrow erosion. Over half a ton of soil has been saved on a variety of soils around the world on a field-by-field basis per ounce of PAM applied. The molecular level PAM's weight, charge density, and charge type all have an impact. PAM's ability to mitigate erosion which is caused by irrigation. For the

best field results, molecular weights of polymers around 6 million are necessary. For rivers and clay-rich soils poor in exchangeable calcium, adding calcium to water, in control or experimental group, can minimize the quantity of soil runoff. Calcium, presumably, stabilizes soil structure by forming ionic bridges.

2.3. The origin of Amino Acids (AAs) in Soil Atmosphere

In soil, nitrogen occurs in organic and inorganic forms. Most surface soils account for greater than 95% of the total nitrogen (Ros *et al.,* 2011). It has been found from literature that about 40% of total nitrogen exists in soils in the form of amino acids. These amino acids are divided into three different categories which are explained as follows:

(i) Free amino acids (FAA): They are present in dissolved form in the soil solution and freely available for plants (Shannon, *et al.*, 2005).

(ii) Exchangeable amino acids (EAA): They are attached with the charged surfaces on soil organic matter and clay particles of soil (Jones *et al.*, 2005).

(iii) Bound amino acids (BAA): The large fractions of amino acids are generally present in the bound form of proteinaceous amino acids in the form of peptides and proteins (Schulten and Schnitzer, 1997).

Bound amino acids (BAA) are mostly available indirectly to the plants and considered as reservoirs. However, the concentration of FAA in soil is found to be in the range of 0-158 μM. Although soil inorganic nitrogen is the significant primary source of plant-nitrogen, soluble organic-nitrogen in soils is generally utilized by plants (Kronzuckerl and Okamotol, 1999). The free amino acid is an important and significant source of energy for soil microorganisms for plant growth, particularly when inorganic-nitrogen availability is limited.

Amino acids in the plants are synthesized by plants themselves. The initial step involves the synthesis of carbon hydrate by means of photosynthesis. This involves the use of carbon, and oxygen from air, hydrogen from water. In the final step, the carbon hydrate is combined with soil's nitrogen, which further results into the amino acid formation via. collateral metabolic pathways. The requirement of amino acids in good enough quantities is known to increase crop yield and their overall quality. The other way by which plants get amino acid, other than the self-synthesis, is direct incorporation of the amino acids into the soil. This facilitate the nutrients supply into the plants and hence improves the microflora of soil as well. Amino acid in the form of liquid and spray provide the efficient building blocks in protein synthesis. Soil thin layer chromatography (soil-TLC) has been studied successfully by various scientists to determine the mobility of amino

acids through pure soil as well as soil amended with WSPs (Mohammad *et al.*, 2012). The determination of mobility of amino acids through soil bed explains the soil-amino acid interactions simultaneously (Tian and Xie, 2008).

Thin layer chromatography (TLC) is a useful analytical method for monitoring and identifying the adsorption behavior of various amino acids using soil as a stationary phase modified with various concentrations of polyvinyl acetate (PVAc). The study uses thin layer chromatography to determine the mobility of four main uncharged amino acids: alanine, tryptophan glycine and glutamine in terms of retention factor. Glycine has the highest mobility of the four amino acids studied. When pure water was utilized as a mobile phase, the order of mobility was determined to be glycine > alanine > tryptophan > glutamine (Khan *et al.*, 2021).

The results show that when the concentration of PVA rises, so does the value of pH. However, the results show that pH and EC are adversely associated. The results also revealed that as the level of PVA in the soil bed increased, the EC decreased (Figure 2). This could be due to differences in the soil's physical structure. The physical and chemical behavior of soil is also influenced by its pH. The electrical conductivity of soil is proportional to salt. The existence of soluble salt in the soil is referred to as salinity. Salt solubility is likely to be affected by soil pH. Soluble salt levels are lower in more alkaline soil.

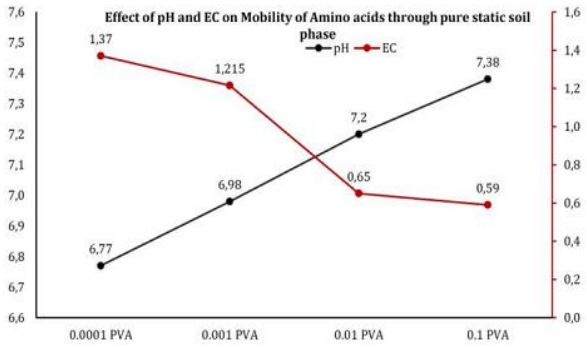

Figure 2. Effect of pH and EC on Mobility of Amino acids amended with various concentration of PVA (0.001%, 0.1%, 1%, 10% respectively) (Khan *et al.*, 2021)

2.4. Research Gap

Polyacrylamide (PAM) as a WSP, has also been used in earlier times by many of the researchers of this field as a soil-conditioner. It has been successfully used to modify the properties of the soil in a better way so that yield of the crops is enhanced in one way or the other. But, the work is majorly

limited to PAM relationship and interaction with soil. Many other WSPs are still in need to explore in this field. So, there is a necessity to discuss and study the relationship between various types of WSPs with soil. In order to inculcate the better understanding of the concepts, different variety of crops can be planted in the amended soil to observe its expected results.

3. Description of Broad Area

Soil chemistry is an interesting area of research. In this area of research, the soil can be amended with various options as discussed above to change its physicochemical properties either in positive or negative way. By looking at the enormous food demand, new agricultural practices will be required to modify the properties of agricultural soil in a better way. Water Soluble Polymers (WSPs) have recently been used in agriculture to demonstrate several benefits, including soil amendments, soil stabilization, water conservation, reduced soil nutrient consumption, curtailing the negative effects of moisture stress in crops and dehydration, minimizing the eutrophication and water contamination caused by fertilizers and pesticides and controlling several phytopathogens. Polyacrylamide (PAM) has been majorly studied by many researchers, as a soil-conditioner, in the earlier times. But many other WSPs are still in need to explore. Amino acids play an integral role in soil chemistry. They can be found in plants directly in the form of nitrogen or can be added indirectly to the plants. The WSPs treated soil would possibly affect the rate of mobility of the amino acids which will ultimately effect the plant growth and crop yield. The focus of this study is to study the direct and indirect effect of the amended soil on plants.

4. Objective of the Research

The objectives of the research are as following:

1. To understand the basic characterizations and concepts of physicochemical properties of the soil.
2. To apply different water soluble polymers on the soil and amend its physicochemical properties.
3. To analyze the changes in physicochemical properties of soil like pH, EC, CEC, mechanical composition, soil structure etc.

4. To evaluate the complex relationship between the soil and amino acids by using soil-TLC (Thin Layer Chromatography) technique.
5. To compose the effectiveness of pure soil and soil amended with WSP on two or more crops.

5. Methodology to be Adopted

The following steps to be followed during research work in order to carry out the proposed research:

1. Collection of soil samples from areas of Aravali and Mirzapur in Faridabad.
2. Laboratory experimental studies which include sieve analysis, moisture content, soil-TLC etc.
3. Characterization techniques including Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), Flame photometry etc.
4. Implementation of amended soil to find out its effects on plant growth and crop production.

6. Expected Outcomes of the Research

The physicochemical properties of the soil taken may possibly fluctuate on amending it with WSPs. The extent of variation would surely depend on soil-type and WSP taken during research. The clay content of soil would affect variations in the properties of a soil as it plays a significant role in the soil-WSP interactions. Mobility of polar and non-polar amino acids may or may not follow a regular trend with the WSP-adsorbed soil as it is again affected by their extent of interactions. One may observe both positive and negative outcomes on plant growth after using the experimental soil.

Proposed Time Frame (Gantt chart)

Year	Plan of Research Work
I	Course work & Literature Survey
II	Visit to areas of Aravali and Mirzapur for soil sample collection and MRIIRS labs for experimental work
III	1. Characterization of the soil samples 2. Communication of the research work
IV	Thesis Writing and Submission

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